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SUMMARY AND REVIEW OF MATERIALS SPECIAL INVESTIGATION GROUP
EVALUATIONS OF HARDWARE FROM THE LONG DURATION EXPOSURE FACILITY

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ABSTRACT

Major materials findings obtained during LDEF post-flight investigations over the past three and one-half years are reported. The summary of findings to date includes results for thermal control coatings, thin polymeric films, composites, metals, adhesives, contamination, and environments definitions. Reaction rates of selected materials exposed to atomic oxygen are presented. Results useful for model verification and comparison with ground based facility data are specifically highlighted. Potential areas for future work are described. In conclusion, a rationale for a second long term flight experiment is presented.

INTRODUCTION

The Materials Special Investigation Group (MSIG), formed by NASA to direct the analysis of materials from the Long Duration Exposure Facility (LDEF), has supported numerous activities since the LDEF retrieval in January 1990. These efforts have included materials analyses, contamination analyses, and definition of the specific LDEF environmental exposure. This exposure over 5.8 years was dominated by ultraviolet radiation (UV) effects at the beginning of the mission. As the orbit decayed near the end of the mission, atomic oxygen (AO) dominated the low Earth orbital effects. Examinations have confirmed the expected performance of selected materials and demonstrated that effects on passive thermal control systems are quite dependent

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upon the exact exposure sequence. The LDEF materials database has supported some important conclusions about engineering uses of selected paints, coated composites, thin polymeric films, adhesives, seals, lubricants, and metals in long term LEO exposures. Detailed reviews of these investigations have been presented at the first three LDEF post-retrieval symposia (refs. 1-3) and two materials workshops (refs. 4-5). Comprehensive reports on these same subjects are nearing completion.

Current (as of November 1993) results of studies of thermal control paints, silverized Teflon™, thin polymeric films, composites, adhesives, lubricants, composites, anodized aluminum, glassy ceramic materials, and effects of contamination on the performance of certain materials are summarized in this paper. This work includes recession rate determinations of some thin polymeric films, silverized Teflon, and composites, embrittlement and/or darkening of thermal control materials under solar exposure, and deposition and subsequent darkening of a number of contaminant films. Some evaluations are extensive and quantitative enough to provide baselines for comparison with ground-based exposure results.

In addition to the extensive investigations already completed, there are still several areas with the potential for providing useful information. Examinations of changes in the thin binder layer in the A-276 paint buttons subsequent to UV exposure will provide information on polymer breakdown mechanisms, structural changes with depth, and clues for manufacturing inherently resistant materials. Selected S13G/LO samples may also be studied in the same manner. UV induced changes in the resin systems of composite materials, Kapton, and Mylar are other subjects which have not been examined in detail. The contamination deposition around vents, near selected outgassing sources, and inside the canisters which opened in stages may be compared to existing contamination models. This activity would require additional analyses to define the amount and nature of the surface deposits in these areas.

ENVIRONMENTS

The values for atomic oxygen and solar UV fluences around the LDEF are reproduced in figure 1. These values include the integrated fluence over all portions of the flight, including the brief exposure in the orbiter cargo bay after retrieval. Detailed information regarding other exposure environments, thermal cycling, meteoroid and space debris impacts, and particulate radiation are found in reports and papers (refs. 6-8) produced by other special investigation groups.

OBSERVED MATERIALS EFFECTS

Thermal Control Paints

White thermal control paints with organic binders retained their optical properties under combined atomic oxygen and UV exposure. However, as the organic binder was eroded by AO, the mechanical integrity of the paint was lost. Trailing edge A-276 specimens, mainly exposed to solar UV and thermal cycling, had a darkened thin outer layer of binder. Specimens taken from

trailing edge locations on the LDEF and reflow on the Evaluation of Oxygen Interaction with Materials-3 experiment (EOIM-3) on the STS-46 shuttle flight partially recovered their initial low absorptance as the damaged binder layer was eroded by atomic oxygen. Exposure to atomic oxygen simultaneous with, or following, solar UV-induced darkening of low absorptivity organic paints can reverse this increased absorptivity leading to performance lifetimes much greater than expected for diffuse paints.

The Z-93 inorganic paint specimens remained white under AO and UV exposure, while the S13G/LO binder darkened, limiting its performance lifetime as the absorptance increased. The black Z-306 paint maintained its optical properties but lost mechanical integrity in the same manner as the A-276. At certain locations on the leading edge the ring of Z-306 surrounding the white A-276 paint buttons was almost completely removed. Z-302 on the leading edge lost binder and pigment essentially down to the substrate. This material retained its absorptance; however, it became diffuse and the emittance was reduced, thus limiting the use of this material for thermal control applications in an AO environment.

Silverized Teflon

The Teflon layer of the nominally 5 mil thick Ag/FEP thermal control blankets used on the A0178 and P0004 experiments recessed under atomic oxygen exposure. Optical property changes as a result of this exposure include an increase in the diffuse component of the reflectance but no change in either solar absorptance or infrared emittance. Specimens from the trailing edge blankets showed no change in their optical properties but did show considerable embrittlement as evidenced by their decreased elongation and ultimate tensile strength values in comparison with leading edge, unexposed, and ground control specimens.

Adhesive backed silverized Teflon tape used on the M0001 experiment, located on the space end of LDEF, ripped along a 90° bend where the tape fastened multi-layer insulation to the aluminum substrate. These failures, shown in figure 2, were most likely caused by the repeated thermal cycling coupled with the associated mechanical motion. The tape was bonded without any allowance for stress relief, and the UV embrittlement lowered the strength of the material. Areas of Ag/FEP bonded to a rigid aluminum substrate, shown in figure 3, increased in solar absorptivity during UV exposure. The application procedure resulted in cracks in the extremely thin (few hundred angstroms) silver and inconel layers. This allowed the adhesive to pass through the metal layers and collect under the FEP layer, where it was darkened by the solar exposure.

Polymeric Films

Thin polymeric films exposed on leading edge locations were eroded significantly and often removed completely under combined AO, solar UV, and thermal cycling exposure. The recession of such films caused failure of the film materials and loss of its engineering function. Failed metallized films produced significant particulate contamination as the loss of the polymer layer allowed the vacuum deposited metals to disperse. Films or coatings which contained inert particle fillers were damaged less severely under the same exposure conditions. This implies that the performance lifetimes of some films may be extended by the atomic oxygen shielding effect of inert particles. Pure organic polymeric films have erosion rates which are linear with exposure to atomic oxygen. This means that the lifetime and performance of such materials is relatively predictable.

Recession rates for Kapton and FEP Teflon, as determined by profilometry, cross-section photomicrographs, or weight change, are given in Table 1.

Composites

Uncoated composite materials eroded under atomic oxygen attack. The organic resins eroded faster than the fibers. Carbon fiber composite erosion rates approached the erosion rate of carbon as the outer resin layer was removed. Recession rates for selected composites are given in Table 1. The recession rate values are averages, since the resins react faster than the carbon fibers. Fiberglass composites became self-protecting as the reactive resin portions were eroded away, exposing the inert glass fibers. As expected, coated composites retained their mechanical properties even when the coatings were degraded over a range of AO and UV exposures. Thin coatings of nickel, SiO₂, and paints all protected composites effectively; no erosion of the coated composites was observed except at meteoroid and space debris impact sites. The significance of these observations is that light-weight composite structures with thin coatings may be used for non-contamination critical LEO applications. Use of such structures in a contamination-sensitive application would be dictated by the properties of the coating material.

Lubricants and Adhesives

Most adhesives and lubricant materials on LDEF were shielded from atomic oxygen and UV radiation and only exposed to vacuum and thermal cycling. Most adhesives used on the LDEF worked well, and post-flight peel strengths were similar to those for control specimens. The LDEF experience verified the use of adhesive tapes for non-structural spacecraft applications. Use of Velcro™ was also verified as an easy method to attach and detach hardware. All Velcro used on LDEF remained functional, although the Dacron thread used to attach Velcro on experiment S1005 failed due to atomic oxygen attack. DC6-1104 RTV adhesive was used successfully to bond Velcro to aluminum and to Z-306 painted blankets. Acrylic adhesive used with UV transparent materials darkened with no change in functional properties. For lubricant materials used on fasteners, the performance was a materials usage issue. The lubricant must be carefully selected if on-orbit cycling is required. Silver plating and MoS₂ on bolts prevented galling (ref. 9).

Chromic Acid Anodized Aluminum

Chromic acid anodized (CAA) aluminum was virtually unchanged by exposure to LEO conditions. This material showed only a slight increase in absorptance at trailing edge locations, attributed to contamination films. CAA aluminum has good potential for long-term space applications. Black chromium plated aluminum, used on experiment A0076 (tray F9) and on Earth end panels, showed large variations in post-flight optical properties. Much of this variation was associated with complex exposure histories, such as shielding by the Earth end support beam and failed aluminum film folding over onto the chromium plating. Figure 4 shows an on-orbit photograph of experiment A0076 and a post-flight close-up of the black chrome plated solar collector.

Metals

Certain metals were affected by the long term exposure to LEO. Atomic oxygen and thermal cycling-induced mechanical stress played major roles in the magnitude of degradation. Thin films of aluminum and tantalum failed under thermal cycling. Leading edge copper straps

darkened, and the absorptance increase correlates with atomic oxygen fluence. The oxidation is superficial; copper would survive if used bare as an interconnect material but would probably operate at a higher temperature over time. Bare 6061-T6 aluminum remained significantly discolored relative to anodized aluminum under both high UV and high AO exposures.

Glassy Ceramics

Glassy ceramics used as mirror coatings increased in film density with reduction in thickness when exposed to AO and solar UV. SiO was converted to SiO₂, and MgF₂ surfaces lost fluorine. These materials had small decreases in solar reflectance. Defects were noted in materials which were not completely oxidized. Coatings for solar mirrors must be selected carefully based on their potential for being oxidized and their susceptibility to densification and reduction in film thickness.

CONTAMINATION

Contamination was present in both particulate and molecular form. A wide variety of particulate contaminants were observed on the LDEF. The distribution of particles include contributions from pre-flight, on-orbit, and post-flight exposures. Debris from failed materials or hardware was transported to other locations during flight, causing large and unpredicted effects in specific areas. The transport mechanisms for this particle movement are not well understood. Particulate debris preferentially collected in the LDEF wake. Aluminizations on thin Mylar and Kapton films created significant particulate contamination after the polymeric material completely eroded away.

Contamination films are common to all materials. Such films may particularly affect short term recession rate measurements. Contaminants may oxidize and act as a sacrificial material to AO erosion or physically block oxygen access to the substrate material. This may be temporary, until the contaminant is oxidized to a volatile species and evaporates, or it may have long term influences if the reaction product, such as SiO_x, is not volatile under the particular flight conditions. Thin molecular film contaminant layers were produced from multiple local sources on the LDEF, including: 1) materials from end structure reflectors, 2) the unbaked Z-306 paint used on the interior of the LDEF structure, and 3) several organic and silicone based materials used on or as part of various experiments. The potential sources and the associated deposited films were subjected to the full range of LDEF exposures depending on their location. The influences of such films on the electrical and optical properties vary with silicone and hydrocarbon sources and exposure. There are a number of competing post-deposition processes which either fix material in place or remove material. Such processes may form films through UV induced reactions which darken and attach material firmly to the substrate or AO oxidation to non-volatile products. Oxidized film layers may also trap materials, allowing subsequent UV darkening of underlying contaminant layers. Silicon was detected at many locations on LDEF but appears to always be associated with a localized source.

MODELING OF SPACE ENVIRONMENT EFFECTS

Figures 5 and 6 show the atomic oxygen fluence vs. time as a percent of total exposure and as a function of incidence angle. The observations of the condition of materials on LDEF are dominated by the fact that the spacecraft experienced over half its oxygen fluence in the last six months of the mission. Materials performance lifetimes are influenced not only by the exposures but the sequence of exposures. The thermal velocity spread among atomic oxygen atoms allows significant amounts of oxygen to reach positions in excess of 100° from the ram direction. Details of the atomic oxygen environment models have been presented in detail in previous reports (ref. 10). LDEF data is available in sufficient amounts to verify atomic oxygen exposure, UV exposure, and contamination deposition models and to predict property changes associated with these types of exposures. Also available are recession rates of FEP, TFE, several composites, and paint systems due to AO exposures, and changes in optical properties due to UV exposures. Using in-flight and post-flight measurements of thermal control coatings, degradation trend analyses were performed for extrapolation of solar absorptance values for Z-93, S13G/LO, A-276 with RTV670 overcoat, A-276 with OI650 overcoat, and chromic acid anodized aluminum (ref. 11). Outgassing of RTV silicones, Z-306 paints, and heat shrink tubing have been measured. Thermal models of the entire spacecraft and selected surfaces have been carried out (ref. 6).

LDEF results allow for many benchmark comparisons with ground-based test facilities. Dimensional changes in various composite laminates due to moisture outgassing in vacuum were measured both during the LDEF flight for the first 370 days and in the laboratory (ref. 12). Diffusion coefficients were also predicted using control and LDEF flight specimens. Atomic oxygen interaction with polymers at defect sites in protective coatings have been studied by Banks, et al. (ref. 13). Using LDEF flight samples and samples exposed in RF plasma ashers in the laboratory, a Monte Carlo model was developed for predicting undercut cavities and atomic oxygen scattering.

Further investigation of simulated space environment, particularly synergistic effects, is needed. Such comparisons could include atomic oxygen recession on composites, Kapton, and FEP Teflon; solar UV darkening of white thermal control paints; mechanical property changes in composites and FEP Teflon including percent elongation to failure and tensile strength; solar cell performance; physical degradation of paints under atomic oxygen exposure; fixing of silicone contaminants on substrates; and specular changes in materials.

RATIONALE FOR FUTURE FLIGHT TESTING

There exists a continuing need for long term LEO exposure testing. Spacecraft missions are being extended to longer and longer times to obtain the maximum return on capital investment, and extended missions cannot rely on design concepts which work for brief exposures. New design concepts must be verified prior to in-service use. The materials flown on LDEF were from the late 1970s and early 1980s, and not all these materials are in current use. Some materials flown which remain in use have been superseded by newer materials with better intrinsic properties but without a flight history. Existing materials must be qualified for longer durations, and new

materials must be qualified where current materials do not suffice. Demonstration of hardware performance identifies any concerns in the development stage and increases the reliability of the spacecraft and its systems. In addition to these program needs, there are supporting technical reasons for flight testing. These included developing the environmental monitoring capability and hardware performance sensors to better define refurbishment schedules, eliminate unnecessary maintenance, and give forewarning of developing hardware or material problems. Flight demonstrations increase the reliability of spacecraft in-service and verify ground-based tests. Flight testing reduces the uncertainty ranges on the engineering design models, leading to increased confidence in the design parameters and reducing risk of performance failure.

Technical considerations for long term test flights include active monitoring of the spacecraft temperatures and ambient environments, and obtaining time-resolved data to separate the ground and on-orbit effects. A polar or near-polar orbit would provide a much higher particulate radiation exposure than was received by the first LDEF, allowing evaluation of materials and systems performance in a new environmental regime. A second extended duration flight would take advantage of a new generation of candidate materials for use as coatings, structural materials, seals, adhesives, optical ports, lubricants, antennas, and communications hardware. Investigations would focus on determination of degradation mechanisms, their temperature dependence, specific exposures, performance characteristics under selected varying environments and the time dependence of property changes. Verification of models for contamination and space debris are high priority items. On-orbit testing would also define acceptable exposure sequencing for combined effects ground based testing. A long term flight test would also qualify accelerated testing methodology for both on-orbit and ground applications.

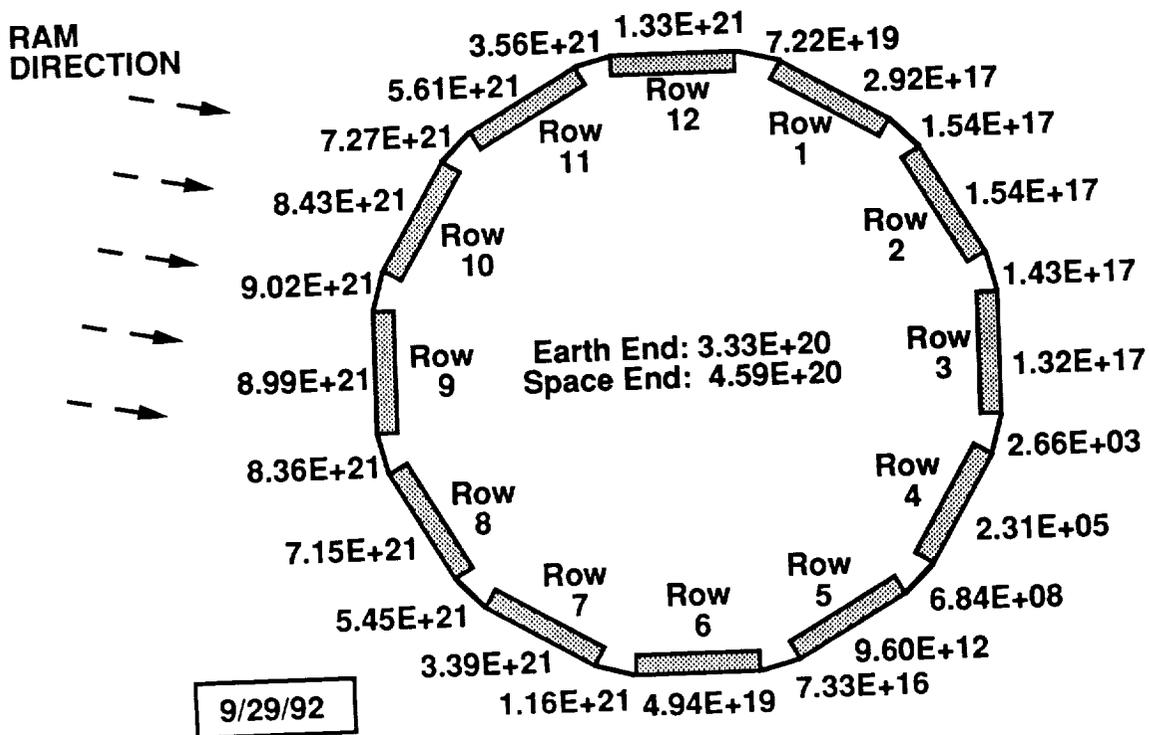
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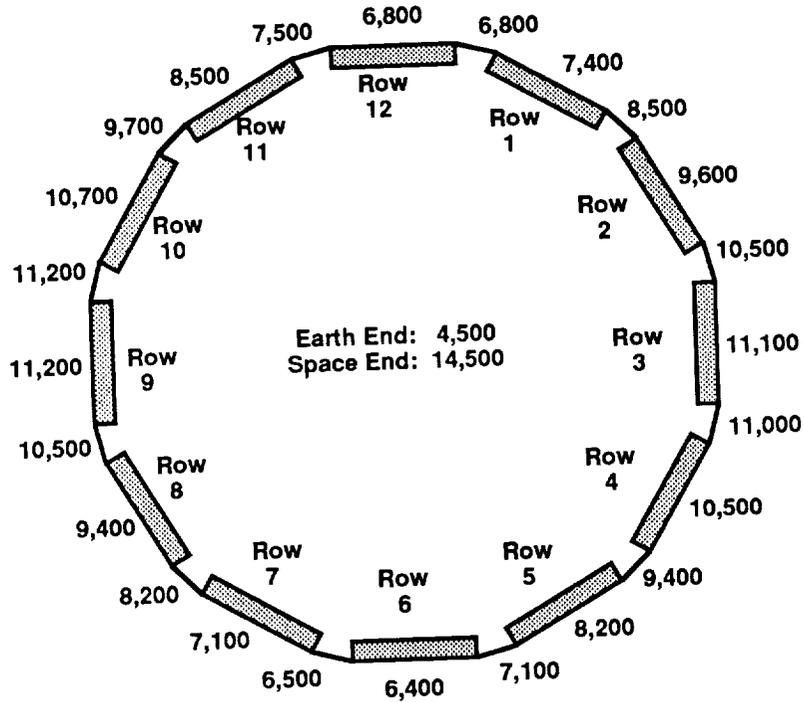
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Table 1. Atomic oxygen induced recession rates.

Composites	R_e (10^{-24} cm ³ /atom)
T300/934 (Graphite/Epoxy)	0.56-1.12
Gr/P1700 (Graphite/Polysulfone)	1.05
Gr/Bismaleimide	1.26
Thin Polymeric Films	
Ag/FEP	0.34
Kapton TM (Space end-Grazing angle incidence)	1.75



ATOMIC OXYGEN FLUENCES (ATOMS/CM²) AT END OF MISSION, INCLUDING EXPOSURE DURING RETRIEVAL



CUMULATIVE EQUIVALENT SUN HOURS EXPOSURE AT END OF MISSION

Figure 1. Mission atomic oxygen and solar exposures for LDEF.



Figure 2. Failure of adhesive backed silverized teflon tape on M0001 experiment located on space end of LDEF.

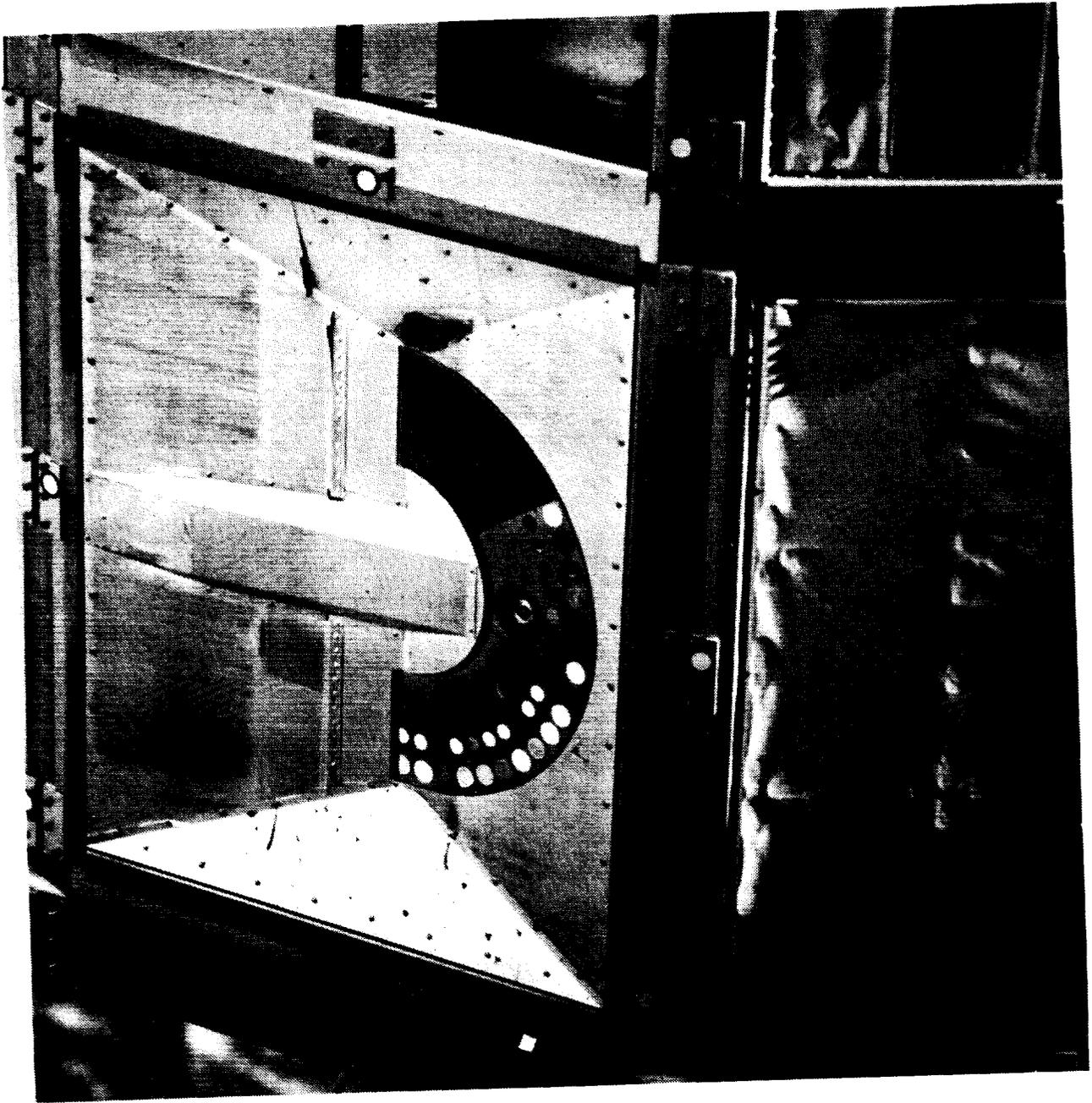


Figure 3. Ag/FEP bonded to rigid aluminum substrate on experiment AO069 located on tray A9.

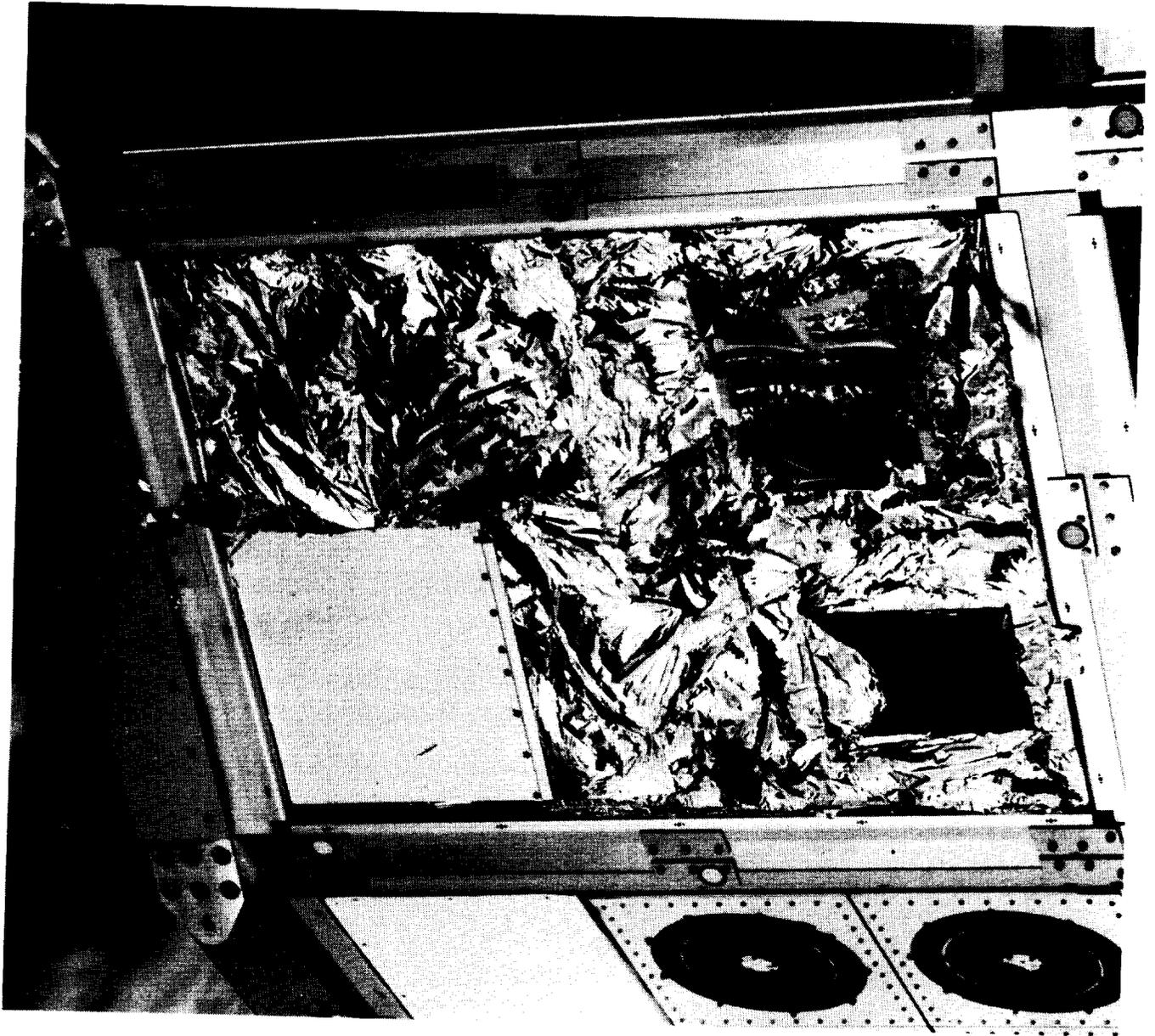


Figure 4a. On-orbit photograph of experiment AO076 showing failed aluminum film folding over chrome plated solar collector.

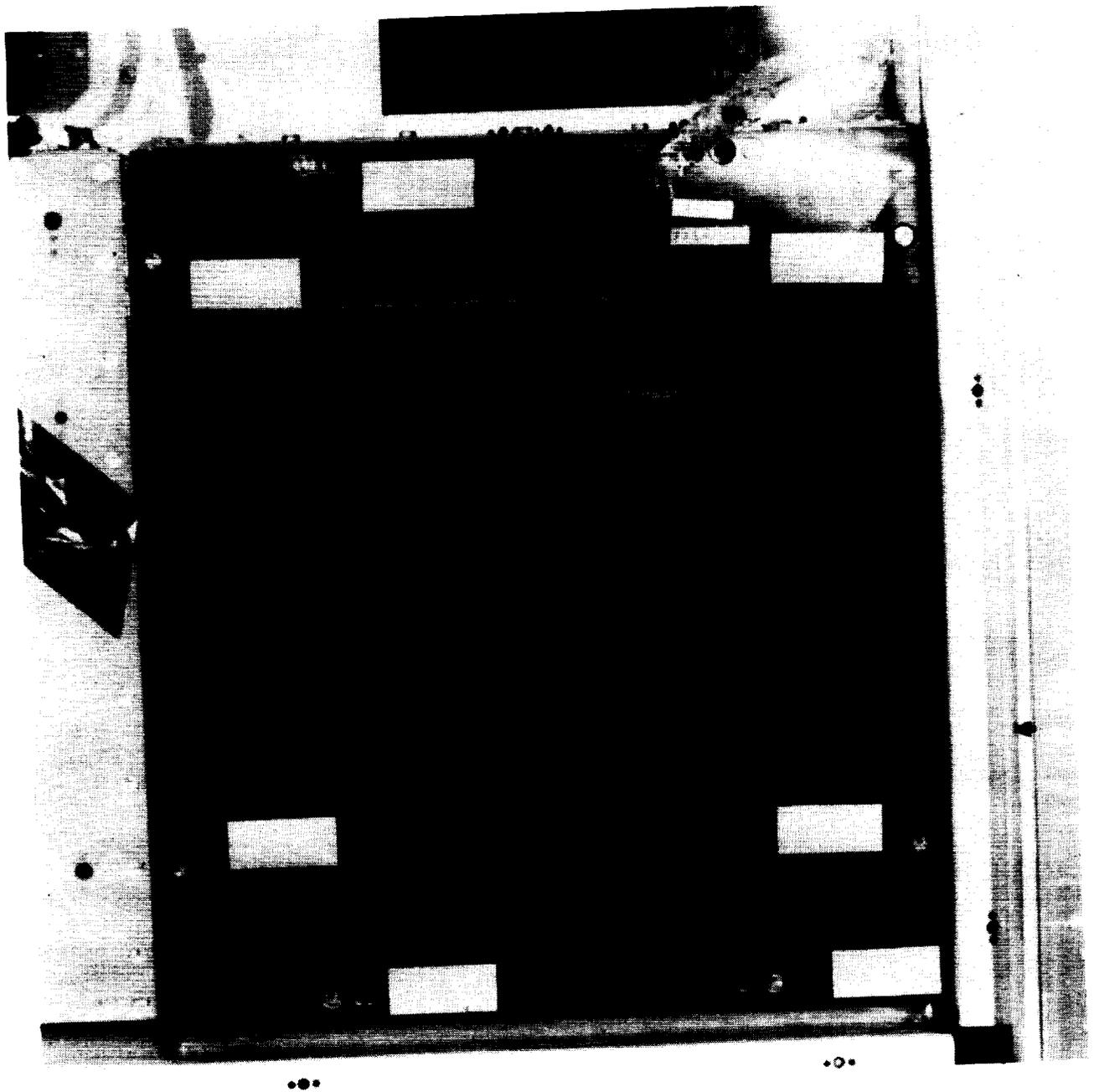


Figure 4b. Post-flight photograph of black chrome plated solar collector.

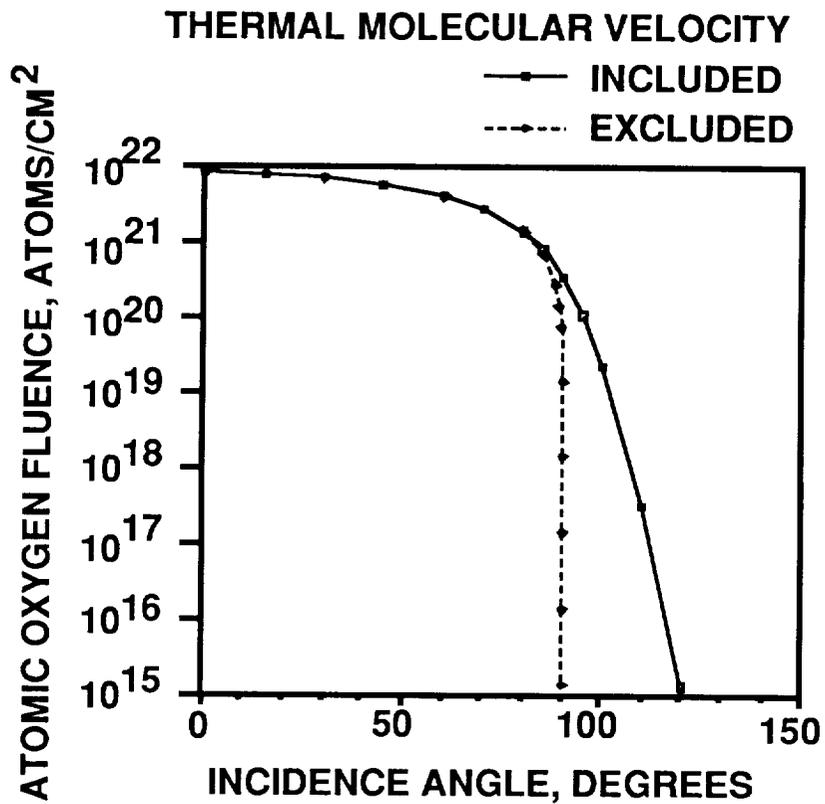


Figure 5. Atomic oxygen fluence as a function of angle of incidence.

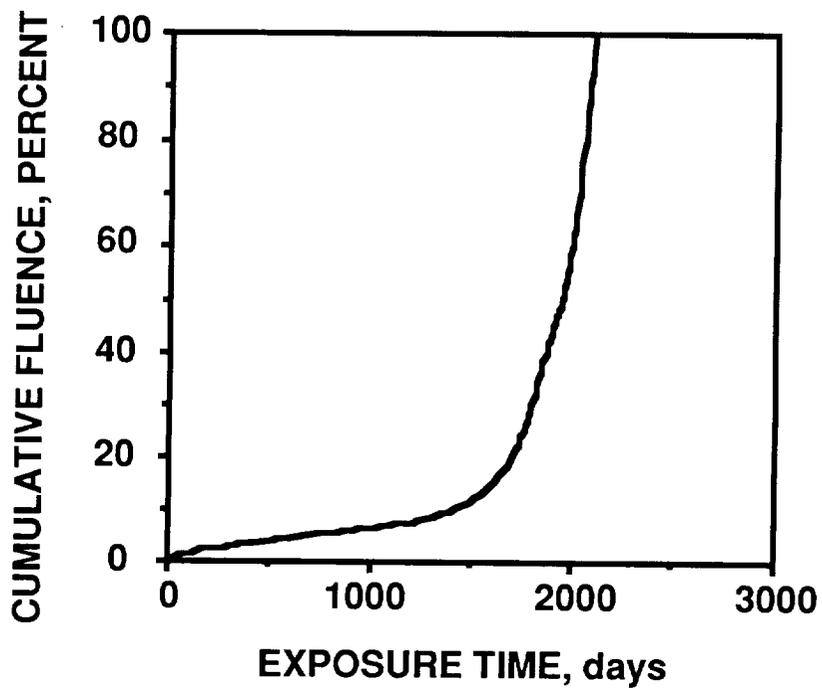


Figure 6. Percent cumulative atomic oxygen fluence as a function of exposure time.

